

FORM PTO-1390
(REV. 11-2000)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371

Mo-6245/WW-5515

U.S. APPLICATION NO. (If known, see 37 CFR 1.5

To Be Assigned 09/787551

INTERNATIONAL APPLICATION NO.

INTERNATIONAL FILING DATE

PRIORITY DATE CLAIMED

PCT/EP99/06576

September 7, 1999

September 19, 1998

TITLE OF INVENTION Aqueous Formulation For Surface Preparation of Paper and Cardboard

APPLICANT(S) FOR DO/EO/US KLOHR, Erik-Andreas; MULLER, Volker; KIESEWETTER, Rene and RAMTHUN, Jurgen

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND or SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. ☒ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☒ is attached hereto (required only if not communicated by the International Bureau).
 - b. ☐ has been communicated by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
 - a. ☐ is attached hereto.
 - b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are attached hereto (required only if not communicated by the International Bureau).
 - b. ☐ have been communicated by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11 to 20 below concern document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☒ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
14. ☐ A **SECOND or SUBSEQUENT** preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
18. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. ☒ Other items or information:

Abstract

To Be Assigned

PCT/EP99/06576

Mo-6245/WW-5515

21. ☒ The following fees are submitted:

CALCULATIONS PTO USE ONLY

BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):

532 Rec'd PCT/PTO 16 MAR 2001

Neither international preliminary examination fee (37 CFR 1.482)
nor international search fee (37 CFR 1.445(a) (2)) paid to USPTO
and International Search Report not prepared by the EPO or JPO \$1000.00

International preliminary examination fee (37 CFR 1.482) not paid to
USPTO but International Search Report prepared by the EPO or JPO \$860.00

International preliminary examination fee (37 CFR 1.482) not paid to USPTO
but international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$710.00

International preliminary examination fee (37 CFR 1.482) paid to USPTO
but all claims did not satisfy provisions of PCT Article 33(l)-(4) \$690.00

International preliminary examination fee (37 CFR 1.482) paid to USPTO
and all claims satisfied provisions of PCT Article 33(1)-(4) \$100.00

ENTER APPROPRIATE BASIC FEE AMOUNT =

\$ 860.00

\$ 0.00

Surcharge of \$130.00 for furnishing the oath or declaration later than ☐ 20 ☐ 30
months from the earliest claimed priority date (37 CFR 1.492(e)).

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	\$
Total claims	14 -20 =	0	x \$18.00	\$ 0.00
Independent claims	2 -3 =	0	x \$80.00	\$ 0.00
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ \$270.00	\$ 0.00
TOTAL OF ABOVE CALCULATIONS =				\$ 860.00
<input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.				\$ 0.00
SUBTOTAL =				\$ 860.00
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				\$ 0.00
TOTAL NATIONAL FEE =				\$ 860.00
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property +				\$ 40.00
TOTAL FEES ENCLOSED =				\$ 900.00
				Amount to be refunded: \$
				charged: \$

a. ☐ A check in the amount of \$ _____ to cover the above fees is enclosed.

b. ☒ Please charge my Deposit Account No. 13-3848 in the amount of \$ 900.00 to cover the above fees.
A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 13-3848. A duplicate copy of this sheet is enclosed.

d. ☐ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO.

James R. Franks
Bayer Corporation
Patent Department
100 Bayer Road
Pittsburgh, PA 15205-9741



00157

PATENT TRADEMARK OFFICE

James R. Franks
SIGNATURE

James R. Franks
NAME

42,552
REGISTRATION NUMBER

03-19-01

PCT

TRANSMITTAL LETTER TO THE
UNITED STATES RECEIVING OFFICE

Date	March 16, 2001
International Application No.	PCT/EP99/0676
Attorney Docket No	Mo-0245/WW-551551

I. ☐ Rectification under 37 CFR 1.10 (if applicable)

532 Rec'd PCT/PTO 16 MAR 2001

EK633384698US
Express Mail mailing number

March 16, 2001
Date of Deposit

I hereby certify that the application/correspondence attached hereto is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to Assistant Commissioner for Patents, Washington, D.C. 20231.

<i>Donna J. Veatch</i>
Signature of person mailing correspondence

Donna J. Veatch
Typed or printed name of person mailing correspondence

II. ☒ New International Application

TITLE Aqueous Formulation For Surface Preparation of Paper and Cardboard

Earliest priority date
(Day/Mon/Year)

(19/09/1998)

SCREENING DISCLOSURE INFORMATION: In order to assist in screening the accompanying international application for purposes of determining whether a license for foreign transmittal should and could be granted and for other purposes, the following information is supplied. (Note: check as many boxes as apply):

- A. ☒ The invention disclosed was not made in the United States.
 B. ☒ There is no prior U.S. application relating to this invention.
 C. ☐ The following prior U.S. application(s) contain subject matter which is related to the invention disclosed in the attached international application. (NOTE: priority to these applications may or may not be claimed on form PCT/RO/101 (Request) and this listing does not constitute a claim for priority.)

application no.		filed on	
application no.		filed on	

- D. ☐ The present international application ☐ contains additional subject matter not found in the prior U.S. application(s) identified in paragraph C. above. The additional subject matter is found on pages and ☐ DOES NOT ALTER ☐ MIGHT BE CONSIDERED TO ALTER the general nature of the invention in a manner which would require the U.S. application to have been made available for inspection by the appropriate defense agencies under 35 U.S.C. 181 and 37 CFR 5.1. See 37 CFR 5.15

III. ☐ A Response to an Invitation from the RO/US. The following document(s) is(are) enclosed:

- A. ☐ A Request for An Extension of Time to File a Response
 B. ☐ A Power of Attorney (General or Regular)
 C. ☐ Replacement pages:

pages		of the request (PCT/RO/101)	pages		of the figures
pages		of the description	pages		of the abstract
pages		of the claims			

- D. ☐ Submission of Priority Documents

Priority document		Priority document	
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- E. ☐ Fees as specified on attached Fee Calculation sheet form PCT/RO/101 annex

IV. ☐ A Request for Rectification under PCT 91 ☐ A Petition ☐ A Sequence Listing DisketteV. ☒ Other (please specify): Preliminary Amendment w/AbstractThe person
signing this
form is the:

<input type="checkbox"/> Applicant	James R. Franks
<input checked="" type="checkbox"/> Attorney/Agent (Reg. No.) 42,552	Typed name of signer
<input type="checkbox"/> Common Representative	<i>James R. Franks</i> Signature

PATENT APPLICATION
Mo-6245
WW-5515

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICATION OF)
ERIK-ANDREAS KLOHR ET AL) PCT/EP99/06576
SERIAL NUMBER: TO BE ASSIGNED)
FILED: HERewith)
TITLE: AQUEOUS FORMULATION FOR)
SURFACE PREPARATION OF)
PAPER AND CARDBOARD)

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Washington, D.C. 20231
Sir:

This preliminary amendment is being filed concurrently with the subject patent application. Upon granting a Serial Number and filing date, please amend the subject patent application as follows.

"Express Mail" mailing label number EK633384698US
Date of Deposit March 16, 2001

I hereby certify that this paper or fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to the Assistant Commissioner of Patents and Trademarks, Washington, D.C. 20231

Donna J. Veatch

(Name of person mailing paper or fee)


Signature of person mailing paper or fee)

Please amend the Application as follows.

IN THE SPECIFICATION:

Please replace the title at the top of page 1 with the following

--AQUEOUS FORMULATION FOR SURFACE PREPARATION OF PAPER AND
CARDBOARD--

Please insert the following at line 3 of page 1 of the specification.

--FIELD OF THE INVENTION--

Please insert the following at line 11, page 1 of the specification.

--BACKGROUND OF THE INVENTION--

Please insert the following at line 18 of page 3 of the specification.

--SUMMARY OF THE INVENTION--

Please insert the following at line 3 of page 4 of the specification.

--DETAILED DESCRIPTION OF THE INVENTION--

IN THE ABSTRACT:

On a separate page, please add the following abstract.

--AQUEOUS FORMULATION FOR SURFACE PREPARATION OF PAPER AND
CARDBOARD

ABSTRACT OF THE DISCLOSURE

Described is an aqueous formulation for the surface finishing of paper and cardboard, which comprises a water-soluble polysaccharide-based cobinder. The cobinder comprises a water-soluble cellulose ether etherified with sulfoalkyl groups.--

A separate abstract page is included herewith.

IN THE CLAIMS:

Please add the following Claims 8 through 14.

--8. The aqueous formulation of Claim 1 wherein said water-soluble cellulose ether is etherified with sulfoethyl groups.

9. The aqueous formulation of Claim 2 wherein the sulfoethylcellulose ether of said cobinder has a degree of substitution by sulfoethyl groups of from 0.2 to 0.9.

10. The aqueous formulation of Claim 1 wherein said cobinder comprises cellulose mixed ethers substituted by sulfoethyl groups to a degree of substitution of from 0.005 to 0.9.

11. The aqueous formulation of Claim 4 wherein the cobinder is selected from carboxymethyl-, methyl-, methylhydroxyethyl-, methylhydroxy-propyl-, hydroxyethyl- or hydroxypropylsulfoethylcellulose with a degree of substitution by sulfoethyl groups of 0.1 to 0.7.

12. The aqueous formulation of Claim 5 wherein said pigment is selected from calcium carbonate, kaolin, gypsum, titanium dioxide and mixtures thereof.

13. The aqueous formulation of Claim 6 wherein the sulfoethylcellulose ether used as cobinder is present in said aqueous formulation at a concentration of from 0.1 to 15%.

14. A substrate selected from paper and cardboard, wherein at least a portion of the surface of said substrate has been finished with an aqueous formulation comprising a water-soluble cellulose ether etherified with sulfoalkyl groups as a polysaccharide-based cobinder.--

Please replace Claim 1 with the following.

1. (Once Amended, Clean) An aqueous formulation for the surface finishing of paper and cardboard comprising a water-soluble cellulose ether etherified with sulfoalkyl groups as a polysaccharide-based cobinder.

Please replace Claim 2 with the following.

2. (Once Amended, Clean) The aqueous formulation of Claim 1 wherein the cobinder comprises a sulfoethylcellulose ether with a degree of substitution by sulfoethyl groups of less than 1.2.

Please replace Claim 3 with the following.

3. (Once Amended, Clean) The aqueous formulation of Claim 2 wherein the cellulose derivative etherified with sulfoethyl groups is selected from carboxymethylsulfoethylcellulose, methylsulfoethylcellulose, methylhydroxyalkylsulfoethylcellulose, methyl-hydroxypropylsulfoethylcellulose, hydroxyethylsulfoethylcellulose and hydroxypropylsulfoethylcellulose.

Please replace Claim 4 with the following.

4. (Once Amended, Clean) The aqueous formulation of Claim 1 wherein the cobinder is selected from sulfoethylcellulose with a degree of substitution by sulfoethyl groups of 0.3 to 0.75; and carboxymethyl-, methyl-, methylhydroxyethyl-, methylhydroxy-propyl-, hydroxyethyl- or hydroxypropylsulfoethylcellulose with a degree of substitution by sulfoethyl groups of 0.01-0.7.

Please replace Claim 5 with the following.

5. (Once Amended, Clean) The aqueous formulation of Claim 1 wherein the formulation further comprises at least one pigment.

Please replace Claim 6 with the following.

6. (Once Amended, Clean) The aqueous formulation of Claim 1 wherein the formulation is free of pigment, and further comprises auxiliary substances.

Please replace Claim 7 with the following.

7. (Once Amended, Clean) The aqueous formulation of Claim 6 wherein the sulfoethylcellulose ether used as cobinder is present in said aqueous formulation at a concentration of 0.1 to 30%.

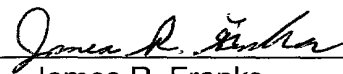
REMARKS

Claims in the case are 1 to 14. Claims 1 to 7 have been amended herein, and Claims 8 to 14 have been added by amendment herein.

Claims 1 to 7 of the above-identified patent application have been amended to place them in better form for examination by the Office. Basis for added Claim 8 is found in original Claim 1. Basis for added Claims 9 and 10 is found in original Claim 2. Basis for added Claim 11 is found in original Claim 4. Basis for added Claim 12 is found in original Claim 5. Basis for added Claim 13 is found in original Claim 7. Basis for added Claim 14 is found at page 1, lines 4-10, and page 3, line 19 through page 4, line 2 of the specification.

The amendments presented herein do not represent the entry of new matter into the application. Applicants respectfully request entry of this amendment.

Respectfully submitted,
ERIK-ANDREAS KLOHR
VOLKHARD MÜLLER
RENÉ KIESEWETTER
JÜRGEN RAMTHUN

By 
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/jme/JRF0039

VERSIONS WITH MARKINGS TO SHOW CHANGES MADE

IN THE SPECIFICATION: (Marked-Up)

The following is a version of the title at the top of page 1, with markings to show changes made thereto in the present Preliminary Amendment.

[Aqueous formulation for the surface finishing of paper and paperboard]

AQUEOUS FORMULATION FOR SURFACE PREPARATION OF PAPER AND CARDBOARD

IN THE CLAIMS: (Marked-Up)

The following are versions of the amended claims with markings to show changes made thereto in the present Preliminary Amendment.

1. (Once Amended, Marked-Up) An [A]aqueous formulation for the surface finishing of paper and cardboard [paperboard with a polysaccharide-based cobinder, characterised in that the formulation contains] comprising a water-soluble cellulose ether etherified with sulfoalkyl groups[, in particular sulfoethyl groups,] as [the] a polysaccharide-based cobinder.

2. (Once Amended, Marked-Up) The [A]aqueous formulation [for the surface coating of paper and paperboard according to] of [c]Claim 1[, characterised in that] wherein the cobinder comprises a sulfoethylcellulose ether with a degree of substitution by sulfoethyl groups of less than 1.2[, preferably of 0.2 to 0.9 for sulfoethylcellulose and 0.005 to 0.9 for mixed ethers].

3. (Once Amended, Marked-Up) The [A]aqueous formulation [for the surface coating of paper and paperboard according to one] of [c]Claim[s] [1 or] 2[, characterised in that] wherein the cellulose derivative etherified with sulfoethyl groups [comprises] is selected from carboxymethylsulfoethylcellulose, methylsulfoethylcellulose, methylhydroxyalkylsulfoethylcellulose, methylhydroxypropyl-

sulfoethylcellulose, hydroxyethylsulfoethylcellulose [or] and hydroxypropylsulfoethylcellulose.

4. (Once Amended, Marked-Up) The [A]aqueous formulation[s for the surface coating of paper and paperboard according to one] of [c]Claim[s] 1 [to 3, characterised in that] wherein the cobinder [comprises] is selected from sulfoethylcellulose with a degree of substitution by sulfoethyl groups of 0.3 to 0.75; [or] and carboxymethyl-, methyl-, methylhydroxyethyl-, methylhydroxy-propyl-, hydroxyethyl- or hydroxypropylsulfoethylcellulose with a degree of substitution by sulfoethyl groups of 0.01-0.7[, in particular of 0.1-0.7].

5. (Once Amended, Marked-Up) The [A]aqueous formulation [for the surface coating of paper and paperboard according to one] of [claims] Claim 1 [to 4, characterised in that] wherein the formulation [contains] further comprises at least one pigment[, in particular calcium carbonate, kaolin, gypsum, titanium dioxide or mixtures thereof].

6. (Once Amended, Marked-Up) The [A]aqueous formulation[s for the surface coating of paper and paperboard according to one] of [claims] Claim 1 [to 4, characterised in that] wherein the formulation [contains no] is free of pigment, and [contains the cobinder alone or together with other] further comprises auxiliary substances.

7. (Once Amended, Marked-Up) The [A]aqueous formulation [for the surface coating of paper and paperboard according to claim] of Claim 6[, characterised in that] wherein the sulfoethylcellulose ether used as cobinder is [used in an aqueous solution with] present in said aqueous formulation at a concentration of 0.1[-] to 30%[, in particular of 0.1-15%].

09/787551

532 Rec'd PCT/PTO 16 MAR 2001

Aqueous formulation for the surface finishing of paper and paperboard

5 This invention relates to the use of water-soluble sulfoethylcellulose ethers, such as for example sulfoethylcellulose ether, carboxymethylsulfoethylcellulose ether, hydroxyethylsulfoethylcellulose ether, methylsulfoethylcellulose ether, hydroxypropylsulfoethylcellulose ether as well as appropriately hydrophobically-modified sulfoethylcellulose mixed ethers as cobinders for aqueous formulations for the surface finishing of paper and paperboard. Such formulations are also known as
10 coating colours.

Water-soluble polyvinyl alcohols, polyacrylamides, polyacrylates, alginates, starches, chitosans, starch ethers, modified starch ethers, nonionic cellulose ethers, such as for example methylcellulose ether, hydroxyethylcellulose ether and hydroxypropyl-
15 cellulose ether as well as hydrophobically-modified cellulose ethers, ionic cellulose ethers, such as for example methyl-, carboxymethylcellulose ether (CMC) and carboxymethylmethylcellulose ether, have long been used as cobinders for pigment-containing coating colours for coating paper and paperboard (c.f. DE 16 21 694, EP 0 399 775, US patent 4 994 112, EP 0 382 576, US patent 5 080 717).

20 The products used as cobinders have the task of controlling the rheological properties required for the coating operation and ensuring uniform coating of the surface of the paper or paperboard. It is known that when cellulose ethers, in particular carboxymethylcellulose ethers, are used, it is possible to establish the required properties for the coating operation by means of the degree of polymerisation of the starting cellulose and the degree of substitution of the cellulose ether.

Apart from establishing the rheological properties determined by the particular application unit, the cobinders in pigment-containing coating colours furthermore have the task of preventing absorption of the coating colour during the coating operation and ensuring unproblematic processing of the colours. Improving water

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Donna J. Veatch

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retention is of particular significance in this connection because it is possible to avoid inadmissible increases in concentration of the coating colour during the coating operation and thus to ensure longer running times or less machine downtime with lower sheet breakage rates. More uniform and thus higher quality coatings may
5 furthermore be ensured as, due to the controlled penetration of the additives present in the coating colours into the base paper during the coating operation and during drying, it is possible to reduce migration of coating colour constituents in the coating (c.f. J.S. Malik, J.E. Kline, Tappi 1992, Coating Conference Proceedings Tappi-Press, Atlanta, pp. 105 to 113).

10 According to the prior art, the rheological properties of coating colours may inter alia purposefully be established by the nature and quantity of pigment (for example chalk and/or kaolin) and of binder or cobinder (for example CMC). When using CMC as cobinder, improved values for the dynamic water release capacity of the coating
15 colour and the quality of the surface coating of the papers may be achieved by a quantitative increase in the cobinder content or by providing a CMC with a higher degree of substitution with carboxymethyl groups of approx. 1 or above. However, these possibilities are associated with economic disadvantages both for the manufacturer of the CMC and for the paper finisher as the improved properties may
20 only be achieved by using larger quantities of raw materials (alkali solution, chloroacetic acid) or by longer reaction times for alkalisation or etherification of the cellulose.

Improved dynamic water release capacity values for the coating colour are also
25 achieved by increasing the molecular weight and viscosity of the CMC used. However, high molecular weight carboxymethylcellulose ethers may only be processed within certain limits as an excessive increase in viscosity of the coating colour must be avoided because processing problems such as coating blade streaks, web breakages and the like may otherwise occur. When high viscosity
30 carboxymethylcellulose ethers are used for coating colours, it is generally only possible to process CMC solutions of a low concentration. However, this approach is

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again associated with impaired quality of the coated papers (for example inter alia reduction in paper whiteness). If high molecular weight cobinders are added in the form of aqueous solutions, this moreover results in the introduction of an undesirably large quantity of water into the pigment coating colour. This results in lower solids contents of the pigment coating colour and extended drying times for the coated paper or paperboard web and thus in higher energy costs for the user combined with lower volume paper throughputs.

The trend towards ever higher machine speeds and the tendency to make increased use, in calcium carbonate, of a pigment, the use of which, when used without the kaolin with which it has hitherto frequently been combined, results in markedly poorer water retention of the coating colour, emphasises the necessity of providing products of an improved quality with a lower dynamic water release capacity. The addition of such an improved cobinder to the pigment coating colour should furthermore not be accompanied by inadmissible increases in viscosity of the colour. The introduction of novel cobinders should furthermore not impair the quality of the coated paper.

It has surprisingly been found that using alkylsulfonated and hydroxyalkylsulfonated carboxypolysaccharides or mixed ethers, such as for example sulfoethylcellulose derivatives, in particular sulfoethylcellulose ethers (SEC), carboxymethylsulfoethylcellulose ethers, methylsulfoethylcellulose ethers, methylhydroxyethylsulfoethylcellulose ethers, methylhydroxypropylsulfoethylcellulose ethers, ethylsulfoethylcellulose ethers, hydroxyethylsulfoethylcellulose ethers and hydroxypropylsulfoethylcellulose ethers as cobinders improves the quality of coating colours. It has been found that, at an identical solids content and identical viscosity, in comparison with coating colours with CMC as cobinder, coating colours which contain cobinders based on sulfoethylcellulose exhibit lower values for dynamic water release capacity, simultaneously combined with improved rheological properties. Moreover, using hydrophobically-modified sulfoethylcellulose mixed

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ethers, such as for example hydroxypropylsulfoethylcellulose ether, results in an improvement in paper quality for printing purposes.

- 5 This effect is inter alia a function of the raw material, of the sulfoalkylation reagent and of the degree of substitution (DS) established by the sulfoalkyl group. Raw materials which are claimed are preferably hetero- and homopolysaccharides, such as for example celluloses (for example chemical cellulosic pulps, linters, crude linters, softwood sulfite, softwood sulfate and/or hardwood pulps), galactomannans (guar, locust bean flour), starches (maize, potato, wheat starches and the like) together with
- 10 the hydrolysates thereof or enzymatically, thermally and/or oxidatively degraded products, pectins, carrageenans, alginates, xanthan, hemicelluloses, chitin and chitosans. Sulfoalkyl-modified proteins, such as for example gelatine and the like, are furthermore also claimed.
- 15 The average molecular weights [M_w] (DAWN four-angle laser light scattering photometer from Wyatt) of the sulfoalkylated polysaccharide ethers claimed according to the invention are at most 10^8 [g·mol⁻¹], preferably at most 200,000 [g·mol⁻¹].
- 20 The sulfoalkyl group-transferring reagents claimed are the compounds conventionally described in the literature, such as for example chloromethylsulfonic acid, chloroethanesulfonic acid, chloropropanesulfonic acid, 1,3-propane sulfone, vinylsulfonic acid or sodium vinylsulfonic acid or the like.
- 25 The sulfoalkylated polysaccharide ethers claimed according to the invention are produced and formulated in the manner generally known to the person skilled in the art. The sulfoalkylated polysaccharide ethers may be added as monosaccharide ethers in powder, pellet or suspension form to the coating colour formulation. For technical or economic reasons, it may be necessary further to react the monoethers in further
- 30 synthesis steps with conventional long- or short-chain alkyl or alkylaryl, carboxyalkyl or hydroxyalkyl group-transferring reagents, such as for example

methyl chloride, ethyl chloride or long-chain glycidyl ethers, chlorinated or brominated hydrocarbons having 3 to 30 carbon atoms; chloroacetic acid or sodium chloroacetic acid; ethylene oxide, propylene oxide or butylene oxide to yield binary or ternary mixed ethers.

5

It is likewise possible to use physical blends of the monoethers claimed according to the invention or of the binary or ternary mixed ethers with conventional, water-soluble unmodified polysaccharides, such as for example starches, alginates or galactomannans, or water-soluble, modified polysaccharides, in particular polysaccharide ethers, such as for example starch ethers (for example carboxymethyl starch, methyl starch, ethyl starch, hydroxyethyl starch, hydroxypropyl starch), cellulose ethers (for example inter alia methylcellulose, ethylcellulose, methylhydroxyalkylcellulose, hydroxyalkylcellulose, carboxymethylcellulose, carboxymethylhydroxyethylcellulose), galactomannan ethers (for example carboxymethyl guar, hydroxypropyl guar).

15

The quantity in which the cobinder claimed according to the invention or the physical blend claimed according to the invention is used in the coating colour formulation is not subject to any limitations. Said quantity is, however, conventionally subject to technical (viscosity) or economic factors. The proportion of cobinder added to the coating colour is conventionally at most 10 parts, relative to 100 parts of pigment, in particular at most 2 parts, preferably at most 1 part.

20

When cellulose-based cobinders are used as an additive for pigment-containing coating colours, the cellulose ethers used are preferably those which have the highest possible solution quality. Gel particles, fibres and specks may result in clogging of filters and screens. If coarser, water-insoluble cellulose particles collect beneath the coating unit, coating blade streaks and web breakage may occur. It is thus necessary to use cellulose ethers which yield aqueous solutions containing no gel particles, fibres or specks. The water-solubility of cellulose ethers is conventionally established by the degree of substitution of the etherification components.

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The term "DS" (degree of substitution) used in relation to the cellulose ethers claimed according to the invention and described by way of example below denotes the average number of substituted hydroxyl groups in the cellulose per anhydroglucose unit. It is advantageous according to the invention if the DS by sulfoethyl groups in the cellulose ethers used is less than 1.2. The person skilled in the art knows that, in order to produce a water-soluble CMC, the degree of substitution must be approx. 0.4 and, in order to produce a water-soluble, no further substituted SEC, the degree of substitution must be at least approx. 0.2-0.3. In the case of aqueous formulations according to the invention comprising SEC without further substituents, the DS of SEC is preferably 0.2 to 0.9, in particular 0.3 to 0.75. If fibre-free solutions are to be produced, the degree of substitution is generally distinctly higher (c.f. EP 0 319 867). In the case of mixed ethers which contain sulfoethyl groups, the degree of substitution by sulfoethyl groups which is necessary to achieve water solubility is distinctly lower. In the case of the mixed ethers used in the formulations according to the invention, the degree of substitution (DS) by sulfoethyl groups is preferably 0.05 to 0.9, in particular 0.01 to 0.8 and particularly preferably 0.1 to 0.7.

The aqueous formulation according to the invention may contain one or more prior art auxiliary agents, preferably from among the group of dispersants (in particular polyphosphates, polyacrylates), binders (in particular starch and starch ethers, casein, polymer dispersions based on butadiene/styrene, acrylic acid ester/styrene, acrylic acid ester/vinyl acetate, vinyl acetate/ethylene and copolymers of the above products with acrylonitrile), antifoaming agents (in particular emulsions of animal or vegetable fats, silicone emulsions or higher alcohols and the esters thereof), optical brighteners and acceptors therefor (in particular polyvinyl alcohols, casein, CMC), shading dyes (in particular pigment dyes, substantive and basic dyes) to adjust the colour location, products to increase water-resistance (in particular melamine and urea resins, glyoxal, epoxy resins) and/or auxiliary agents which are required for the final finishing of the coated paper (in particular calcium, sodium or ammonium

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stearate, wax dispersions, polyglycols and polyethylene dispersions). Pigments used in pigment-containing coating colours are the raw materials familiar to the person skilled in the art, such as kaolin, natural calcium carbonate, talcum, satin white, precipitated calcium carbonate, titanium dioxide, aluminium hydroxide, gypsum, barium sulfate, plastic pigments alone or blended in dry or already predispersed ("slurry") form.

In another embodiment of the invention, the aqueous formulation is a so-called sizing agent, which contains the cobinder in a pigment-free formulation, optionally together with one or more of the above-stated auxiliary agents. Such a formulation preferably comprises an aqueous solution which contains the sulfoethylcellulose ethers in a concentration of 0.1 to 30%, in particular 0.1 to 15%.

The cobinders used in the Examples were added as highly concentrated, aqueous solutions of the pigment slurry. The aqueous solutions of the sulfoethylcellulose derivatives claimed according to the invention all have elevated transmission values of $T > 95\%$ (2 wt.% aqueous solution, wavelength of light used $\lambda = 550$ nm, optical pathlength of cell = 10 mm) [Hitachi model 101 spectrophotometer, Hitachi Ltd., Tokyo, Japan]. In this manner, low fibre contents of $< 1\%$ may be ensured.

A dynamic measurement method, as described in the literature (c.f. Das Papier 50 (1996), no. 3, 97 et seq.), is used to determine dynamic water release, rheological properties and immobilisation of the coating colours.

The cellulose ethers shown in Table 1 are used for the tests described below. Production of the sulfoethylcellulose derivatives claimed according to the invention is known from the literature (c.f. US-A 2 811 519; US patent 4 972 007; EP 0 319 865 A2).

Table 1: Characteristics of products used to determine water retention

No.	Sample no.	Product	Viscosity [mPa.s] ¹⁾	MS ²⁾	DS ²⁾	pH ³⁾	Comment
1	Walocel CRT 5 G ⁴⁾	Carboxymethyl- cellulose	990		0.77	10.3	Reference sample
2	SEC 1	Sulfoethylcellulose	805	-	0.70	8.6	Invention
3	SEC 2	Sulfoethylcellulose	843	-	0.48	7.0	Invention
4	SEC 3	Sulfoethylcellulose	823	-	0.49	6.3	Invention
5	SEC 4	Sulfoethylcellulose	1408	-	0.41	6.9	Invention
6	SE guar	Sulfoethyl guar	1294	-	0.49	7.5	-
7	CM guar	Carboxymethyl guar	1542	-	0.59	6.4	Reference sample for guar ethers
8	HPSE guar	Hydroxypropylsulfo- ethyl guar	814	0.95	0.1	8.3	-
9	HPSEC	Hydroxypropylsulfo- ethylcellulose	852	2.0	0.18	6.0	-

- 1) Brookfield RVT, 100 rpm, T = 25°C, absolutely dry, nominal viscosity range:
800-1600 mPa.s (c = 10%) for Walocel CRT 5 G and corresponding samples
- 2) Average degree of substitution by sulfoethyl, carboxymethyl [DS] and
hydroxypropyl groups [MS]
- 3) pH value of 10%, aqueous solutions
- 4) Commercial product of Wolff Walsrode AG

The products described in Table 1 are incorporated into a coating colour with kaolin and chalk as pigment in accordance with the standard formulation shown in Table 2. Rheological behaviour and initial water release are investigated using a dynamic measurement method. The formulation shown in Table 2 is initially used as the standard formulation. There is, however, no consequent restriction to the formulation ingredients stated therein.

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Table 2: Standard coating colour formulation for investigation of water release capacity

Formulation ingredients	Solids content [%]	Quantity in colour [g]
Kaolin ¹⁾	99	30
Chalk ²⁾	99	70
Latex binder ³⁾	50	12
Cobinder (c.f. Table 1)	99.5	0.3
Optical brightener ⁴⁾	100	0.35
Glazing auxiliary ⁵⁾	50	0.75
Solids content established with water: 66% to 71% (c.f. Table 3)		
pH of the colours established with sodium hydroxide solution: 8.5-9.5		

- 5
- 1) Amazon 88, Kaolin International B.V., Netherlands
 - 2) Hydrocarb 90, Omya, Germany and Plüss Staufer, Switzerland
 - 3) Baystal P 8588, Polymer Latex GmbH, Germany
 - 4) Blankophor P, liquid, Bayer AG, Germany
 - 5) Calcium stearate, Henkel KGaA, Germany

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Table 3 shows the results from the investigation of rheological properties and initial water release capacity under dynamic measurement conditions, in each case with 0.3 parts of cobinder relative to 100 parts of pigment.

Table 3: Comparison of rheological properties and initial water release capacity (WRC) of coating colours

No.	Sample ¹⁾	SC [%] Colour ²⁾	Cobinder content ³⁾	Viscosity 1 [mPa.s] ⁴⁾	Visco- sity 2 [mPa.s] ⁵⁾	Immobilisation		Dynam. WRC at 5 bar ⁶⁾ [mg/min x cm ²]
						Time [s]	SC [%]	
1	without cobinder	69.8	0	580	666	10	70.3	>100
2	Walocel CRT 5 G (blank sample)	69.8	0.5	615	691	105	71.1	50
3	SEC 1	69.8	0.5	492	428	60	71.0	46
4	SEC 4	69.8	0.5	474	418	60	71.1	48
5	SEC 3	69.8	0.5	564	499	60	70.9	49
6	SEC 2	69.8	0.5	492	421	60	71.0	42
7	without cobinder	71.1	0	1002	1443	10	71.5	>100
8	Walocel CRT 5 G (blank sample)	71.2	0.5	1050	1585	240	73.1	46
9	SEC 1	71.2	0.5	703	629	50	72	36
10	Walocel CRT 5 G (blank sample)	66.2	1.2	420	391	20	67.0	45
11	SE guar	66.4	1.2	291	276	30	67.2	40
12	HPSE guar	66.2	1.2	279	258	30	67.4	42
13	CM guar (blank sample)	66.4	1.2	354	337	30	67.1	58
14	HPSEC	66.4	1.2	347	311	20	67.2	43

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- 1) Sample names c.f. Table 1
 - 2) Solids content of colour
 - 3) Cobinder content in the formulation shown in Table 2
 - 4) Low-Shear Haake CV 100, start-up viscosity at a shear rate of gamma point =
5 300 1/s
 - 5) Low-Shear Haake CV 100, viscosity after 10 minutes' shear at a shear rate of
gamma point = 300 1/s
 - 6) Water release capacity (WRC) at 5 bar at time $t = 0$ min [= immediate]
- 10 In principle, the tendency when producing coating printing papers is towards using
coating colours having the lowest possible water content, i.e. a high solids content.
As a result, it is possible to achieve energy savings during drying, higher throughputs
and more voluminous coating films. In order to ensure flowability of such a colour,
the viscosity and water release, in particular the initial water release, of a coating
15 colour should be low. In this way, it is possible to minimise the risk of a sudden
increase in viscosity and thus of the solids content of the coating colour during the
coating operation. If the water retention of the colour is within a critical range, the
pressure impulses acting beneath the coating unit of approx. 2-4 bar may result in the
solids content beneath the coating blade rising abruptly due to the rapid penetration
20 of the water into the paper. This results in "boiling over" of the scraper or in beard
formation, i.e. deposits of the coating colour on the coating unit, coating blade
streaks or paper web breakages.
- After the coating operation, immobilisation of the coating colour means that all
25 transport processes of formulation ingredients present in the formulation are stopped.
If this operation lasts too long, migration processes may be initiated both in the paper
and at the paper surface which, under certain circumstances, may result in quality
defects on printing (for example mottling).
- 30 A dynamic measurement method, as described in the literature (c.f. D. W. Jones et al.
in: *Das Papier*, 50 (3) (1996) pp. 97 - 106), was used in the tests performed in this

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case to investigate water retention in coating colours. In this method, the coating colour to be tested is introduced into the above-stated measurement cell and subjected to shear under defined conditions. The released quantity of water is here recorded as a function of the solids content instantaneously present in the colour. The value for the released quantity of water at time $t = 0$ is designated the initial water release. The results in Table 3 show that the coating colours formulated with sulfoethylcellulose ethers (nos. 3-6) exhibit improved dynamic water release capacity at 5 bar in comparison with the standard (Walocel CRT 5 G (= prior art)). In comparison with coating colours formulated with CMC, the addition of sulfoethylated polysaccharides, such as for example sulfoethylcellulose, hydroxypropylsulfoethylcellulose, sulfoethyl guar, hydroxypropylsulfoethyl guar (c.f. Table 3), surprisingly gives rise, inter alia, to a liquefying action in the coating colour. The water retention of the products remains at a constantly high level. It is striking that the measured viscosities are still distinctly below those of the unthickened coating colour (c.f. Table 3, no. 1 with no. 3 to 6 and no. 7 with no. 9). This results in the above-stated advantages, in particular in highly pigmented, particularly critical formulations, as shown in nos. 7-9. Example no. Despite the similar level of viscosity of the aqueous solutions (c.f. Table 4), the viscosity of the coating colour thickened with SEC is distinctly below the level of the reference sample and of the unthickened colour.

Moreover, in contrast with the reference sample (Walocel CRT 5 G [Table 3, nos. 2 and 8]), immobilisation of the colour is more rapid when coating colours formulated with sulfoethylcellulose ethers are used. The risk of migration processes, for example of binder fractions at the coating surface, is accordingly minimised.

Machine tests were performed on a coating plant using the products shown in Table 4.

Table 4: Characteristics of the cobinders used for machine tests

No.	Sample	Viscosity [mPa.s] ¹⁾	DS-SE ²⁾	Salt content [%]	pH
1	Walocel CRT 5 G (CMC) ⁴⁾ (blank sample)	990	0.77	<0.5	10.3
2	Walocel CRT 3 G (CMC) ⁴⁾ (blank sample)	406	0.82	<1	7.7
3	SEC 1 ⁵⁾	805	0.70	<1	8.6
4	Hydroxypropylsulfoethyl- cellulose ether	363	0.2 [MS: 2.1]	<1	6.0
5	SEC 6 ⁵⁾	323	0.75	<0.5	6.6

¹⁾ Brookfield RVT, 100 rpm, T = 25°C, absolutely dry, nominal viscosity: 800-1600 mPa.s (nos. 1 & 3) or 300-450 mPa.s (nos. 2, 4, 5)

²⁾ Degree of substitution by sulfoethyl or hydroxypropyl groups (MS)

³⁾ pH of 10% solutions

⁴⁾ Commercial product of Wolff Walsrode AG

⁵⁾ Sulfoethylcellulose ether

The standards used in this case were the products Walocel CRT 5 G and Walocel CRT 3 G (both carboxymethylcellulose ethers). The standards were investigated in comparison with sulfoethylcellulose ethers (SEC 1 and 6) and hydroxypropylsulfoethylcellulose ether (HPSEC). The cobinders were incorporated in the standard formulation stated in Table 5 into an already predispersed pigment slurry with the additives stated below.

The base paper used was a ligneous LWC paper (36 g/m²). In each case, approx. 9 g/m² of coating colour were applied onto the surface of the paper at coating machine speeds of approx. 1600 m/min. The coating was applied to both sides (screen upper

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side first, then screen side) using a coating blade system (stiff blade). There is, however, no consequent restriction to such application systems. Other application units, such as for example roller, jet or spray units may, in principle, be used.

- 5 The coated papers were then dried and glazed and printed by the web offset process. Table 6 shows the results for rheological properties, immobilisation and dynamic water release capacity.

Table 5: Machine tests / Formulation¹⁾

No.	Formulation ⁵⁾	SC ²⁾ [%]	Test number ³⁾				
			V1	V2	V3	V5	V6
1	Hydrocarb 90 MHH	78	70	70	70	70	70
2	Amazon 88	72	30	30	30	30	30
3	Baystal P 8588	50	12	12	12	12	12
4	Walocel CRT 5 G	10	0.50	-	-	-	-
5	SEC 1	10	-	0.50	-	-	-
6	HPSEC ²⁾	10	-	-	0.50	-	-
7	Walocel CRT 3 G	14	-	-	-	0.50	-
8	SEC 6	14	-	-	-	-	0.50
9	Blankophor P	100	0.35	0.35	0.35	0.35	0.35
10	Calcium stearate	50	0.75	0.75	0.75	0.75	0.75
Total			113.6	113.6	113.8	113.6	113.6
Nominal SC ⁴⁾			69.0%				
pH value established:			8.5 - 9.0				

- 5 ¹⁾ Nominal speed: 1600 m/min
Application rate for upper side and screen side: 9 g/m²
Moisture content for upper side and screen side: 4 and 5.5% respectively
Blade (SB): 0.457/40° (tip 3°)
Drying profile, IR/AIF: pause after 1st IR (+ falling AIF)
- 10 Calender: 600 m/min; nip: 11, N/mm: 160, temperature (H1/H2): 90/90°C
- ²⁾ SC ≡ solids content which was established in every case initially for the screen upper side
- ³⁾ V1 contains Walocel CRT 5 G (standard) compared with V2 (SEC 1 as cobinder); V5 contains Walocel CRT 3 G (standard) compared with V3 (HPSEC) and V6 (SEC 6 as cobinder)
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- 4) SC \equiv solids content which was established in every case initially for the screen upper side
- 5) Formulation ingredients were purchased from the manufacturers shown in Table 2.

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Unlike in the preliminary testing to determine water release capacity under dynamic measurement conditions, the aim of the practical tests was to formulate coating colours having the highest possible solids content so as to be able to assess running behaviour in the coating operation under realistic conditions. The attempt was initially made to apply the coating colours onto the surface of the paper at a solids content of 69.0%. On coating the screen upper side, which was performed first, the solids content had to be reduced during the test as rheological problems occurred due to "boiling over" of the scraper and the like. The results for coating the screen side (SS) should accordingly be used for the purposes of comparing the samples with each other (Table 6).

Table 6: Results with regard to rheological properties and dynamic water release capacity (WRC) of the coating colours used for machine testing

No	Sample ¹⁾		SC ²⁾	Difference	Visco-	Visco-	Immobilisation		Dynam. WRC
			[%]	in SC ³⁾	sity 1 ³⁾	sity 2 ³⁾	Time	SC	at 3 bar
				[%]	[mPa.s]	[mPa.s]	[s]	[%]	[mg/min . cm ²] ⁵⁾
1	Walocel CRT 5 G (reference sample)	V1B SS	65.7	-	204	189	130	67	39
2	SEC 1	V2 SS	67.3	+ 1.6	187	176	60	68	55
3	Walocel CRT 3 G (reference sample)	V5 SS	65.4	-	182	175	105	68	50
4	HPSEC	V3 SS	67.1	+ 1.7	204	195	90	68	45
5	SEC 6	V6 SS	67.3	+ 1.9	225	221	78	66	53

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- 1) Sample see Table 5; SS = screen side
- 2) Solids content of colour
- 3) Low-Shear Haake CV 100, start-up viscosity at gamma point = 300 1/s
- 4) Low-Shear Haake CV 100, viscosity after 10 minutes' shear at gamma point = 300 1/s
- 5) Water release capacity (WRC) at 3 bar at time t = 0
- 6) Difference in solids content relative to reference sample

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All solids content (SC), pH value and viscosity data relate to the final value (\equiv screen side) on the application unit.

The results of the preliminary tests, in which sulfoethylcellulose derivatives were associated with lower thickening performance or stronger liquefying effects, are

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- confirmed. The results of the tests performed with SEC no. 1, SEC no. 6 and HPSEC (c.f. Table 6, nos. 2, 4, 5) show that approx. 1.5-2% higher solids contents may be established in every case. The colour viscosities obtained were virtually identical in comparison with the relevant reference samples (c.f. Table 6, nos. 1 and 3). While
- 5 dynamic water release capacity at 3 bar reveals similar values in every case, the use of coating colours thickened with sulfoethylcellulose derivatives is associated with distinctly shorter immobilisation times. Reference has already been made to the consequent advantages which arise during drying or subsequent coating.
- 10 While at the start of the coating tests the solids contents had to be reduced by adding water in order to avoid problems during application due to the formation of deposits on the application unit (for example "boiling over" of the scraper, stalagmite formation), application of the colour to the screen side was characterised by more uniform running behaviour of the paper web. The solids contents for the coating tests
- 15 on the screen side remained virtually constant throughout the test (c.f. Table 7, comparison of start and end).

Table 7: Machine tests³⁾ / screen side results ¹⁾ (= end of test)

No.	Cobinder	Test	Solids contents [%]		Viscosity ²⁾ [mPa.s]		Applica- tion rate [g/m ²]	Moisture content [%]
			Start	End	Start	End		
1	Walocel CRT 5 G (reference sample)	V1B	66.1	66.0	380	370	9.0	5.5
2	SEC 1	V2	67.5	67.3	330	350	9.0	5.4
3	Walocel CRT 3 G (reference sample)	V5	65.9	65.4	390	360	9.3	5.3
4	HPSEC	V3	67.2	67.1	370	390	8.9	5.4
5	LP-S-32172	V6	67.4	67.3	470	460	9.3	5.6

¹⁾ Results for screen side (= end of test)

5 pH values of colours in every case 8.6-8.8; c.f. no. V1B (blank sample) with V2; V5 (blank sample) with V3 and V6

²⁾ Viscosity by Brookfield RVT, 100 rpm, T = 28°C

³⁾ Coating machine speed: 1600 m/min

10 Tables 8 and 9 show the results of paper testing on uncalendered and calendered paper.

Table 8: Results of paper tests on uncalendered paper/VESTRA (screen side) (\equiv end of test)¹⁾

Standard test	Method	V1B	V2	V3	V5	V6
Basis weight [g/m ²]	DIN ISO 536	57.1	57.6	57.3	57.9	58.0
Whiteness, R457 [%]	DIN 53145 I/2	78.2	78.1	78.2	78.5	78.1
Thickness [μ m]	DIN EN 20534	68	68	67	68	68
Yellowness	DIN 6167	4.30	4.35	4.64	4.26	4.31
Opacity [%]	DIN 53 146	92.59	92.47	92.43	92.95	92.56
Colorimetry L*	DIN 5033	91.83	91.78	91.95	91.97	91.80
a*		- 0.70	- 0.70	- 0.70	- 0.60	- 0.68
b*		2.45	2.51	2.67	2.42	2.48
Specific volume [cm ³ /g]	DIN 53 105	1.19	1.17	1.17	1.17	1.17
Sheet density [g/cm ³]	DIN EN 20 534	0.84	0.85	0.85	0.85	0.86
Roughness [μ m]	DIN ISO 8791-4	4.51	4.37	4.11	4.31	4.12
Tappi 75° gloss [%]	Tappi 480 OM 92	18.5	19.0	18.8	18.4	18.9

- 5 Results for screen side (\equiv end of test), c.f. no. V1B (blank sample) with V2; V5 (blank sample) with V3 and V6

Table 9: Results of paper tests on calendered paper/VESTRA (screen side) (\equiv end of test) ¹⁾

Standard test	Method	V1B	V2	V3	V5	V6
Basis weight [g/m ²]	DIN ISO 536	57.4	56.9	56.5	56.7	56.4
Whiteness, R457 [%]	DIN 53145 I/2	76.2	76.0	75.8	76.3	76.2
Thickness [μ m]	DIN EN 20534	52	52	53	51	51
Yellowness	DIN 6167	4.93	5.13	5.49	5.13	5.03
Opacity [%]	DIN 53 146	91.29	91.42	91.14	91.57	90.99
Colorimetry	DIN 5033	91.10	91.05	91.07	91.16	91.08
L*		- 0.78	- 0.78	- 0.88	- 0.74	- 0.78
a*		2.82	2.90	3.15	2.91	2.87
b*						
Specific volume [cm ³ /g]	DIN 53 105	0.90	0.92	0.93	0.90	0.90
Sheet density [g/cm ³]	DIN EN 20 534	1.11	1.09	1.07	1.11	1.11
Roughness [μ m]	DIN ISO 8791-4	1.39	1.54	1.47	1.45	1.37
Tappi 75° gloss [%]	Tappi 480 OM 92	58.8	56.9	54.0	55.4	57.2

- 5 ¹⁾ Results for screen side (\equiv end of test), c.f. no. V1B (blank sample) with V2; V5 (blank sample) with V3 and V6

The differences between the samples are in every case within the standard deviations applicable to the paper tests.

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The coating colours formulated with different cobinders or papers coated therewith were then printed by the web offset process. Table 10 shows the results from the printability tests.

Table 10: Results of offset proofs

Test	Paper sample ¹⁾				
	V1B	V2	V3	V5	V6
Print gloss [%]	26	24	23	26	24
Optical density [%]	1.45	1.45	1.39	1.43	1.42
Dry picking [cm/s] ²⁾	isolated	isolated	isolated	isolated	isolated
Wet picking [cm/s]	isolated	isolated	isolated	isolated	isolated
Absorption test ³⁾	300	300	300	300 - 600	300
Mottling test ⁴⁾	2	1	0	2	1
Blister test	no blisters	no blisters	no blisters	no blisters	no blisters

- 5 1) Results screen side; ink: 408020; comparison to be made: V1B (standard) with V6; V3 with V5 (standard) and V6
- 2) Ink: 408002
- 3) Ink: 520068
- 4) Ink: 408010; 0 = no mottling, 1 = little mottling, 2 = slight mottling
- 10 5) Web offset, 54 g/m² both sides coated and glazed, web width: 0.57 m (size 76 cores), type: Rotoman C (MAN), speed: 25,000 sheets/h (approx. 4.4 m/s), dryer length 10 m (3 sections), inking units: 4, recto and reprint, colour sequence: black/cyan/magenta/yellow (Huber standard LWC inks [Michael Huber GmbH, Munich]), dampening agent composition: isopropanol content
- 15 13% (pH value = 4.8) (dampening agent additive Hydrofix A 8085-09, 2%)

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5 With regard to gloss, values of at least 23% and at most 26% are obtained for the screen side. Optical density values are within the range from 1.39 to 1.45 and thus exhibit only slight variation. For all papers, standard ink no. 408002 causes break-up of individual coating particles. In contrast, the softer ink numbered 408001 does not pick. The picking resistance of the papers is in the critical range in every case. It is, however, not possible to differentiate between the samples, as they are all at a uniform quality level. All papers exhibit rapid absorption behaviour, which has a positive impact on ink drying.

10 Printing results were assessed particularly positively in tests V2 and V6. In particular, the colour (V3) formulated with hydroxypropylsulfoethylcellulose exhibits advantages on printing.

15 Within the conditions set for the tests (web temperature 120-150°C), there were no instances in any of the tests of blistering, the term used to describe the sudden escape of steam from the surface of the coating or the base paper.

Patent Claims

1. Aqueous formulation for the surface finishing of paper and paperboard with a polysaccharide-based cobinder, characterised in that the formulation contains
5 a water-soluble cellulose ether etherified with sulfoalkyl groups, in particular sulfoethyl groups, as the cobinder.
2. Aqueous formulation for the surface coating of paper and paperboard according to claim 1, characterised in that the cobinder comprises a
10 sulfoethylcellulose ether with a degree of substitution by sulfoethyl groups of less than 1.2, preferably of 0.2 to 0.9 for sulfoethylcellulose and 0.005 to 0.9 for mixed ethers.
3. Aqueous formulation for the surface coating of paper and paperboard according to one of claims 1 or 2, characterised in that the cellulose derivative
15 etherified with sulfoethyl groups comprises carboxymethylsulfoethylcellulose, methylsulfoethylcellulose, methylhydroxyalkylsulfoethylcellulose, methylhydroxypropylsulfoethylcellulose, hydroxyethylsulfoethylcellulose or hydroxypropylsulfoethylcellulose.
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4. Aqueous formulations for the surface coating of paper and paperboard according to one of claims 1 to 3, characterised in that the cobinder comprises
25 sulfoethylcellulose with a degree of substitution by sulfoethyl groups of 0.3 to 0.75 or carboxymethyl-, methyl-, methylhydroxyethyl-, methylhydroxypropyl-, hydroxyethyl- or hydroxypropylsulfoethylcellulose with a degree of substitution by sulfoethyl groups of 0.01-0.7, in particular of 0.1-0.7.
5. Aqueous formulation for the surface coating of paper and paperboard according to one of claims 1 to 4, characterised in that the formulation
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contains at least one pigment, in particular calcium carbonate, kaolin, gypsum, titanium dioxide or mixtures thereof.

- 5 6. Aqueous formulations for the surface coating of paper and paperboard according to one of claims 1 to 4, characterised in that the formulation contains no pigment and contains the cobinder alone or together with other auxiliary substances.
- 10 7. Aqueous formulation for the surface coating of paper and paperboard according to claim 6, characterised in that the sulfoethylcellulose ether used as cobinder is used in an aqueous solution with a concentration of 0.1-30%, in particular of 0.1-15%.

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AQUEOUS FORMULATION FOR SURFACE PREPARATION
OF PAPER AND CARDBOARD

ABSTRACT OF THE DISCLOSURE

Described is an aqueous formulation for the surface finishing of paper and cardboard, which comprises a water-soluble polysaccharide-based cobinder. The cobinder comprises a water-soluble cellulose ether etherified with sulfoalkyl groups.

COMBINED DECLARATION AND POWER OF ATTORNEY

ATTORNEY DOCKET NO

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name. I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

AQUEOUS FORMULATION FOR SURFACE PREPARATION OF PAPER AND CARDBOARD

the specification of which is attached hereto,

or was filed on **September 7, 1999**

as a PCT Application Serial No. **PCT/EP99/06576**

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s), the priority(ies) of which is/are to be claimed:

198 42 962.2
(Number)

Germany
(Country)

September 19, 1998
(Month/Day/Year Filed)

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose the material information as defined in Title 37, Code of Federal Regulations, §1.56 which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.)

(Filing Date)

(Status)

(patented, pending, abandoned)

(Application Serial No.)

(Filing Date)

(Status)

(patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

WW 5515-US

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

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